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TRANSMISSION ELECTRON MICROSCOPY OF B-NANOPARTICLES IN HEXANE SOLUTION

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14. ABSTRACT

Transmission electron microscopy revealed b-nanoparticles of 25 to 100 μm. The hexagonal network and cluster of these particles are also obtained at higher magnification. Raman spectral analyses of b-nanoparticles confirmed the spectral peaks are not from hexane solvent, but from b-nanoparticles in hexane solution.

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b-nanoparticles Raman spectroscopy Electron microscope

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INTRODUCTION

Boron is attractive as a fuel or a fuel supplement in propellants and explosives due to high heats of combustion (ref. 1). Technical data is currently available for combustion characteristics of large boron particles, but very little exists for nano-sized boron. Previously, boron nanoparticles were developed by gas-phase decomposition of diborane, which is a highly toxic and flammable gas. In the new method developed by Philip Power and colleagues (ref. 2) at the University of California, Davis does not use flammable gases and it is carried out at the room temperature. The boron tribromide goes through the reduction process using naphthalenide in dry dimethoxyethane. This process produces a bromide-capped intermediate that reacts with octanol to form the first organo capped boron nanoparticles. Different capping agents can be used to obtain this kind of capped boron nanoparticles. The transmission electron microscopy (TEM) revealed bright field TEM images of boron nanoparticles. The TEM is one of the key analytical tools of nanoscience. Since it is not possible to detect boron by energy dispersive x-ray analyses, Raman spectroscopy was conducted to confirm the presence of boron. Raman spectroscopy generally determines the strength of the covalent bonds. The structure model of broad boron sheets and boron nanotubes has good electronic and mechanical properties and is analogous to a single graphite sheet (ref. 3).

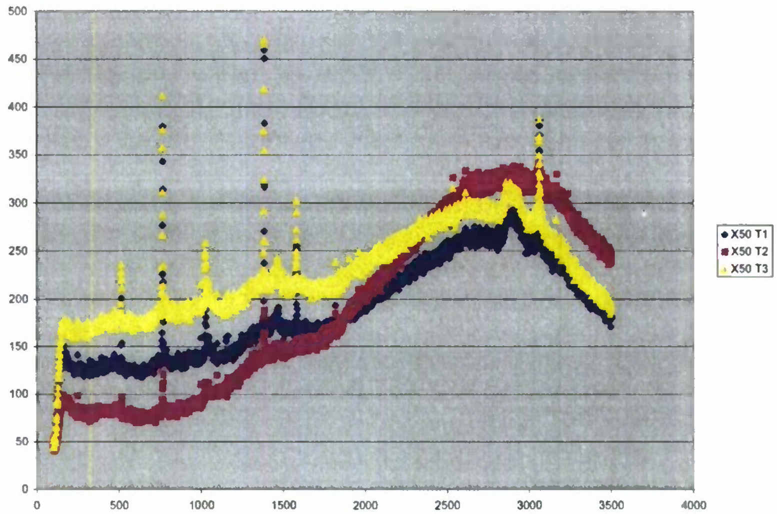
SPECIMEN PREPARATION

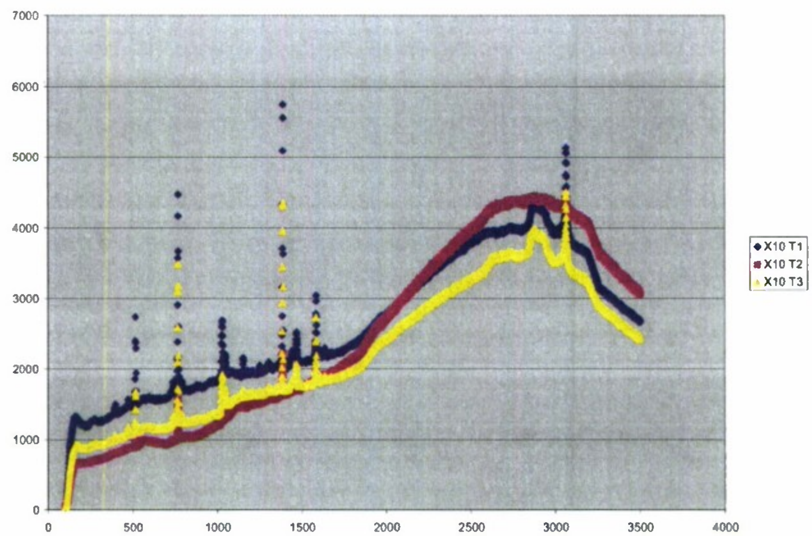
The boron nanoparticles are in hexane solution and they are capped by octanol in which H has been removed. Thus boron nanoparticles are bonded to OR groups (B-octyloxy)n.
A small amount of this solution was dropped on a 200-mesh carbon coated copper grid and dried at room temperature. This specimen was then placed in a single tilt specimen holder and inserted in a Philips 420 TEM.

RESULTS AND DISCUSSION

Raman spectroscopy from hexane solvent did not show any peaks. Either this hexane solution evaporates in the laser or it does not have a strong Raman response. Therefore it is concluded all the peaks observed in Raman spectrums (fig. 1) taken from boron nanoparticles in hexane solution are entirely from boron nanoparticles. Raman Spectroscopy was run three times for sample on two different magnifications, 10X and 50X. In these results a total of six different runs, the major sharp peaks are usually at the same spots. Those broad peaks seem to appear at greater concentration of material, which are probably due to the boron nano-particles from very large chunks.

Electron micrograph in figure 2 shows a bright field TEM image at 15,000X magnification. The hexagonal network structure consisting of b-nanoparticles is indicated by arrow A with each nanoparticles (dark dots) sitting on the lattice points of the hexagonal (0001) plane. The theoretically predicted crystal structure of boron nanomaterial is expected to be orthorhombic, tetragonal, or rhombohedral, which can be a hexagonal structure by crystallographic transformation process. It is possible hexane may be adsorbed onto the surface or in the pores of the b-nanomaterials. Even though the hexane has dried at high temperature, it takes more energy to remove a hexane molecule in a small pore or crevice than it does if it just sits on a surface. This is due to adsorbate-adsorbent interactions, which are different for each system. Also, as mentioned earlier, Raman spectral analyses suggest the same kind of adsorbate-adsorbent interaction takes place in hexane. The area B indicates a large number of b-nanoparticles bounded by two single walled construction of nanotube.The size of a single nanoparticle indicated by C is approximately 250 Å. The electron micrograph in figure 3 taken from different area at magnification 42,000X shows a cluster of b-nanoparticles indicated by A. The broad peak in Raman spectrum is due to the presence of these cluster materials. Electron micrograph in figure 4 shows the dispersed nanoparticles as indicated by A and B at magnification 42,000X. The particle size is approximately 250 Å. The number of particles in area B is larger than that in area A.





Results from Raman Spectroscopy of Boron Nanoparticles Suspended in Hexane											
X10T1			X10T2		X10T3		X50T1		X50T2		X50T3
2840.11	4085.75	2840.11	4401.75	2840.11	3693.75	2840.11	267.25	2840.1	327.5	2840.11	302.25
2840.81	4056.75	2840.81	4409.75	2840.81	3703.75	2840.81	270.25	2840.78	332	2840.81	298.75
2841.5	4038.75	2841.5	4418.25	2841.5	3706.75	2841.5	268.25	2841.49	320	2841.5	297.75
2842.2	4072.25	2842.2	4409.25	2842.2	3679.75	2842.2	272.75	2842.19	333	2842.2	290.25
2842.89	4083.75	2842.89	4388.75	2842.89	3676.75	2842.89	265.25	2842.87	337.5	2842.89	299.25

Figure 1
Raman spectral analysis of b-nanoparticles in hexane

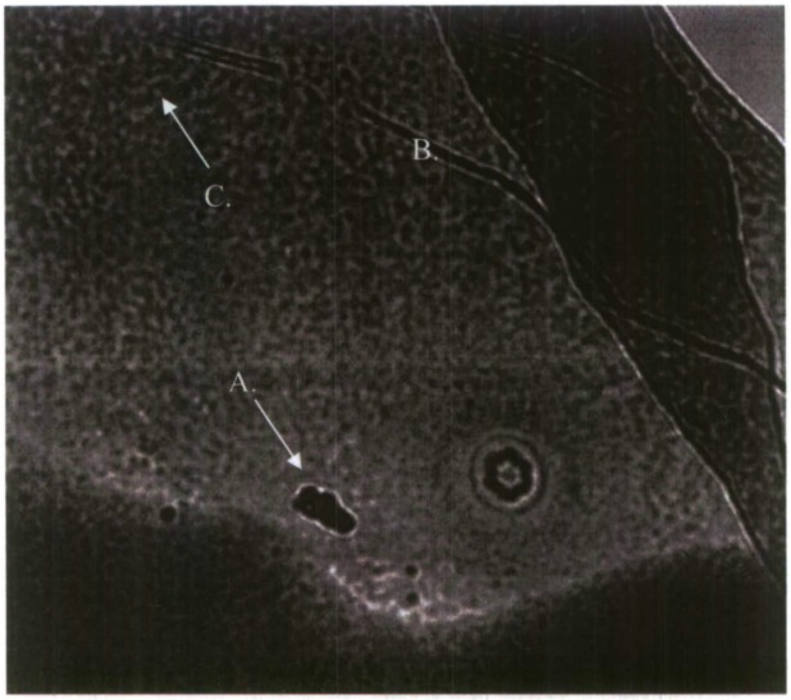


Figure 2
Electron micrograph of b-nanoparticles at 15,000X showing the hexagonal network

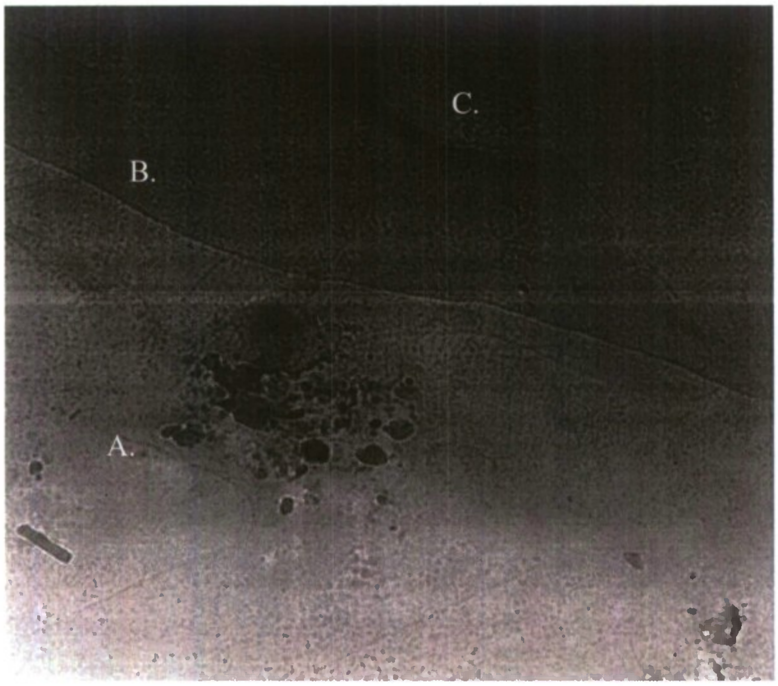


Figure 3
Electron micrograph of b-nanoparticles at 42,000X showing cluster of particles

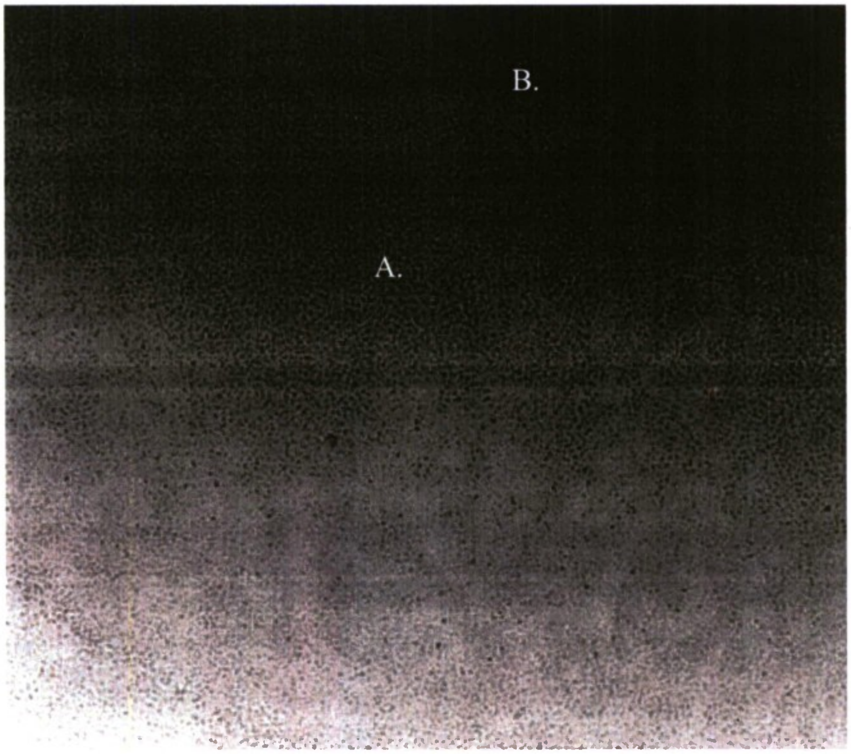


Figure 4
Electron micrograph of b-nanoparticles at 42,000X showing dispersed nanoparticles

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